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PARTICLE SIZE DISTRIBUTION BY
ELECTRICAL SENSING-ZONE COUNTER

Charles M. Stone, Lt, USAF

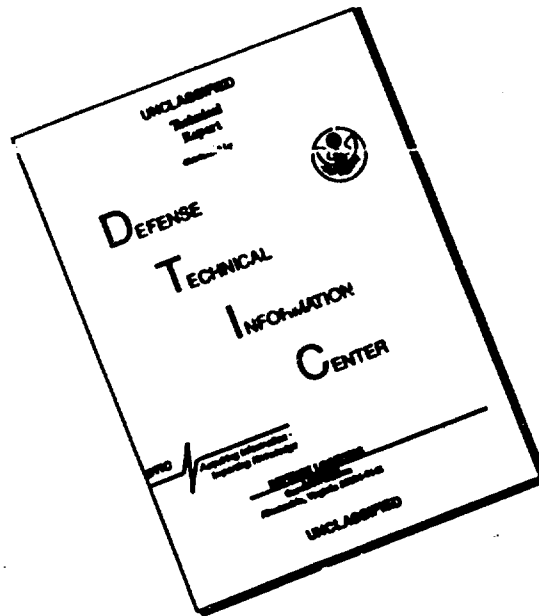
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November 1966

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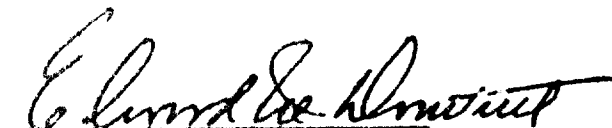


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FOREWORD

The work described herein was performed in the Chemical and Materials Branch of the Propellant Division at the Air Force Rocket Propulsion Laboratory, during the period August 1964 to June 1965. The work was done under Project 3148 by Charles M. Stone, Lt, USAF, Project Engineer. The author acknowledges the editorial and technical assistance given by Dr. Lawrence P. Quinn and Mr. Robert A. Biggers, microscopy assistance by Mrs. Elizabeth Dalaba, and particle counter setup and operating assistance by Mr. Mike Citro.

This report has been reviewed and approved.



ELWOOD M. DOUTHETT

Colonel, USAF

Commander, Air Force Rocket Propulsion Laboratory

ABSTRACT

The objective of this study was to evaluate a method for the quality control of fine powders as an alternate method to that specified in MIL-P-27412 (USAF). The size distribution of various particles in the 3-100 micron range was studied using a counter which operates by the volumetric displacement of an electrolyte in an electrical sensing zone. The particle size distribution is affected by its compatibility with the electrolyte solution, and technique used to prepare the particles for sampling. Data obtained for size distribution was compared with data obtained by microscopic scanning, and from manufacturer's specifications.

Analyses of aluminum powder, coke flour, and hydraulic oil, as well as the calibration sample ragweed pollen are given.

Results show that the electrical sensing-zone counter can be used to give size distributions of a large number of 3-100 micron size particles with a good degree of accuracy, repeatability and speed.

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PARTICLE SIZE DISTRIBUTION BY ELECTRICAL SENSING-ZONE COUNTER

INTRODUCTION.

The particle size distribution determination outlined in this report is offered as an alternate method for the quality control of aluminum powders in accordance with MIL-P-27412 (USAF). The determination of particle size distribution using an electrical sensing-zone apparatus has been in use for several years for sizing of various types of microscopic dusts, emulsions, fibers, contaminants, and related particles. The primary limitation of the method is in finding suitable media for the particles.

Many laboratories have used the counter on a variety of particles but the published information is limited. O. A. Ullrich¹ gives a comparison of particle-sizing methods with special emphasis on the electrical sensing-zone principle. His information comes from work done at Battelle Memorial Institute for size distribution studies of sand, lime, glass beads, mica, iron, uranium oxide, fuel oil, blood cells, pollens, and other materials. Ullrich comments that the electrical sensing-zone method is good for high-speed counting, and high accuracy. Robert H. Berg² in his paper "Electronic Size Analysis of Sub-sieve Particles by Flowing Through a Small Liquid Resistor" also stated this method checks closely with other methods.

The studies described in this report cover size distributions of coke flour in aqueous solution, aluminum dust in aqueous and non-aqueous solutions, and attempts to work with contaminant particles in hydraulic oil. The coke flour and aluminum dust results show that the electrical sensing-zone method is very useful and quite accurate when a large number of particles are available for sizing. Solvation

problems developed while preparing the hydraulic oil for testing. The non-polar oil and non-polar solvents would mix readily but when this mixture was added to the polar electrolyte an emulsion or precipitate would form and hence make it unsuitable for use in the electrical sensing-zone counter.

THEORETICAL BACKGROUND AND INSTRUMENTATION.

A Coulter Counter Industrial Model A was used for all data gathered. Data is obtained by forcing particles suspended in an electrically conductive liquid through a small aperture having an immersed electrode on both sides. As a particle passes through the aperture, the resistance between the electrodes changes. This produces a voltage pulse of short duration having a magnitude proportional to particle volume. The voltage pulses are amplified and fed into a threshold circuit having an adjustable threshold level. If this level is reached or exceeded by a pulse, the pulse is counted. The pulse and threshold level are indicated on an oscilloscope screen thus enabling the operator to visually adjust to a desired threshold level.

The volume of solution flowing through the aperture is controlled by a vacuum-operated manometer connected to the aperture tube. The manometer contains mercury and start-stop current probes. The external vacuum siphons the mercury past all the stop contacts and just past the start contact. The polarity switch is then reversed, starting the current flowing through the electrolyte containing the particles, at the same time the vacuum is removed. As the mercury starts returning to its normal level it first hits a start connection, starting the electronic scaling of the particles. Stop probes are placed so that precisely 50, 500, or 2000 microliters of electrolyte are drawn through the aperture, and the particles contained are electronically scaled and counted.

CALIBRATION AND CALCULATIONS.

With each change of aperture tube or electrolyte solution there is a corresponding change in the resistance between the electrodes. A calibration must therefore be made for each new aperture tube and each new electrolyte solution. Mono-sized particles were used for calibration. In this case ragweed pollen having a nearly constant spherical diameter of 19.5 microns was used in a .9% NaCl-H₂O electrolyte. Knowing the average size of the mono-sized particles and adjusting the threshold level just to the top of the voltage pulses on the oscilloscope screen, the calibration constant (k) can be determined. The calibration constant (k) is derived from $k = \frac{d}{\sqrt{t}}$ where (d) is equal to the average diameter of the mono-sized particles and (t) is equal to the threshold value (t') times a factor (F). The factor (F) is the corrected aperture current setting determined from the resistance of the electrolyte solution. Once the calibration constant is determined, the threshold value (t') and the aperture current (I) can be changed manually, thus giving different size levels at which particles can be counted. The size range can also be expanded by changing a gain index (G) which changes the current flow by a factor of $\sqrt{2}$. The corresponding counts (n) for different sized particles can be put on a cumulative weight percent against the particle size range as is done in Graph I for ragweed pollen. The size distribution of the particles can then be determined from the graph.

A factor which could affect the accuracy of the particle sizing is the passage of more than one particle through the aperture orifice at one time which may be counted as one particle larger than either real one, or a large particle may overshadow a smaller one. This is corrected by a coincidence factor (p) $p = 2.5(D/100)^3 (500/V)^3$. In this equation (D) is the aperture diameter in microns and (V) is the volume of electrolyte measured in microliters. No appreciable error is encountered when the coincidence correction factor is less than 10% of the particles counted at each size level.

The largest diameter particle in a sample should be between 30% and 40% of the orifice diameter for maximum accuracy.

There are a number of errors that can be introduced by the operator but with good techniques and clean apparatus most of these errors can be eliminated.

RESULTS.

a. Ragweed Pollen.

A size-distribution test was made on the ragweed pollen after the calibration had been completed. The data (Table I) were computed and graphed on log-probability paper (Figure 1). Figure 1 shows that about 88% of the pollen are between 18 and 22 microns in diameter, with the median at 19.5 microns. These results were checked by scanning the pollen with a monocular microscope equipped with a calibrated filar eye piece and a 10X objective lens. The eye piece was adjusted to resolve particles 19.5 microns in diameter, and a random scanning of particles on filter paper was performed. The particles measured were filtered from the electrolyte solution that had been used to gather data for Figure 1. The results of the microscopic scanning showed that approximately 90% of the particles were between 18 and 22 microns compared with 88% by the Coulter Counter.

b. Coke Flour.

Two samples of coke flour, 3020 and 3015, were obtained from Great Lakes Carbon Corporation. Data from a lab control report (Tables IIb, IIIb) using a sieve analysis method was used as a check for the electrical sensing-zone apparatus.

Ten milliliters of isopropyl alcohol were used to disperse .001 \pm .0002 grams of coke flour, and the resulting mixture was added to approximately 240 milliliters of .9% NaCl-H₂O electrolyte. A 200-micron aperture tube was used, and 2000 microliters of electrolyte were displaced during each counting period.

Graphing particle diameter and cumulative weight percent on log-probability paper showed sample 3015 to have a greater number of large particles than sample 3020 (Figure 2). Approximately 90% of the particles in 3020 were smaller than 74 microns where as in sample 3015 only 74% were smaller than 74 microns. The sieve analysis gave a similar result, although the percent of small particles was greater than the electrical sensing-zone method. This can be seen by comparing Tables IIa, IIb, IIIa, and IIIb.

A count was taken at the same dial setting before and after the counting period. This enabled the operator to determine whether or not any settling, or chemical reactions with the electrolyte media had occurred. Evidence of settling was observed in a trial count of the coke flour so the counting was set up to count the large particles first, thus correcting for the settling. A stirring propeller with adjustable speed was used to agitate the solution. If stirring is not adequate to keep the large heavy particles suspended, the viscosity of the electrolyte may be increased to help support the particles. This is done by dissolving small amounts of glycerol, sugar, or any other thickening agent in the electrolyte. Thickening agents that do not interfere with the particle counting must be selected.

c. Aluminum Powder.

A sample of Reynolds 1-131 aluminum powder was tested for size distribution. The particles were measured in .9% NaCl-H₂O electrolyte and in 4% NH₄CNS-isopropyl alcohol. Because aluminum powder reacts with an aqueous solution, both an aqueous and a non-aqueous electrolyte were tried in order to compare the degree of reaction and the possible effect of the aqueous solution on the accuracy. The same procedure described earlier of mixing and dispersing of particles was used.

A smaller aperture tube (140 micron) and smaller measuring volume (500 microliters) were used for the aluminum because the largest particle observed while scanning with the microscope was 20-25 microns.

There was evidence of settling, agglomeration or reaction in the .9% NaCl-H₂O electrolyte solution when a trial count was made. In this case the small particles were measured first since they would dissolve first. With the 4% NH₄CNS-isopropyl alcohol electrolyte, the solution was more viscous and the aluminum particles did not appear to settle or react with the solution. This was shown by consecutive counts taken at the same level at the start, middle, and end of the counting period. A slight difference in the end points of the two curves can be noted when comparing Tables IV and V, and Figures 3 and 4 of the particles in different media. The aqueous media data shows fewer small particles and more large particles. This would be typical of initial reaction of small particles followed by a slow agglomeration of particles near the end of the counting period. However, when one graph was overlaid upon the other, the major portion of the curves were superimposed. Repeated tests showed this to be true. Hence either medium could be used if the counting period is not longer than 15-20 minutes. The median diameter of the Al particles is about 10, 5 microns for both the aqueous and non-aqueous solutions.

These results were checked by visually counting approximately 500 aluminum particles under a microscope and arranging them into groups. A sample of aluminum ($.001 \pm .0002$ grams) was first dispersed with 10 ml of isopropanol, added to 240 ml of 4% NH₄CNS-isopropanol electrolyte, and the mixture filtered through 0.8-micron filter paper. After adding immersion oil to make the filter paper transparent, the particles were counted under a monocular microscope using a 43X objective lens and a filar eye piece. The counts were put on a cumulative weight percent basis (Table VI) (use of cumulative

volume or size would give the same results), and plotted against particle size on log-probability paper. A plot was made for both a small number of points as well as for a larger number of points, and a plot of the counter data superimposed for comparison. Figure V shows that the microscope and counter are quite close but the curve for the microscope has a steeper, but not significantly different, slope.

d. Hydraulic Oil.

Hydraulic oil MIL-H-5606 was tested for particle contamination. The hydraulic oil was mixed with various "coupling" solvents and electrolyte solutions in varying concentrations (Table VII). Some of these mixture ratios were taken from Section 12 of the instruction manual for the Coulter Counter, the remainder were devised by the author.

A large amount of oil was needed because of the small amount of particle contamination. This poses a problem because the high resistivity of the non-polar oil makes it unusable with the resistance principles of the Coulter Counter. The non-polar "coupling" solvent also has a high resistance. To lower the resistivity to an acceptable level the oil and "coupling" solvent were mixed with polar electrolytes of low resistivity. The resulting mixtures were tested for resistivity using a standard electrode probe. Since a .9% NaCl-H₂O electrolyte gives a complete range of settings on the counter, the amounts of non-polar and polar liquids were varied until the resistance was about the same as the .9% NaCl-H₂O electrolyte. By varying the concentrations of solvent and electrolyte, the oil concentration was varied between 20% and 50%. Because of the small amount of particle contamination the percent of oil was kept above 20%.

No satisfactory results were obtained from any of the mixtures. Either an emulsion or precipitate formed and hence made the mixtures unsuitable for measurements with the Coulter Counter. Speed and

simplicity are desired and because the particle contamination is very low it is much faster to use microscopy methods.

CONCLUSIONS.

The electrical sensing-zone Coulter Counter can be used to obtain size distribution of particles in the 2 to 100 micron range and can probably be extended in both directions with the use of proper aperture tubes. This analytical technique has been primarily used for fine dry particles that are random samples of larger quantities. The results of the counter check closely with results obtained by sieve screen and microscopy techniques. The data graphed on probability-logarithmic paper (K-E 359-24) can be used in various ways according to the needs of the individual.

This method is much faster than microscopy when a large number of particles are to be counted. But when the total particle count is less than 500 particles, microscopic methods are more expeditious.

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1. Ullrich, O. A., "Size Analysis of Fine Particles and Results Obtained with an Electrical Sensing-Zone Particle Analyzer", Instrument Society of America Conference (1960).
2. Berg, Robert H., "Electronic Size Analysis of Subsieve Particles by Flowing Through a Small Liquid Resistor", ASTM Special Technical Publication No. 234, (1958), pp 245-258.
3. Coulter Counter Industrial Model A Instruction Manual (1963), Section 6, 9.

TABLE I
RAGWEED POLLEN

Sample - Ragweed Pollen
Electrolyte - .9% NaCl-H₂O
Wt of Sample - Not Taken

Aperture - 140 μ
Measuring Vol - 500 μ l
Total Vol - 250ml

| <u>n</u> | <u>d</u> | <u>% Above Stated Size</u> |
|----------|----------|--------------------------------|
| 1422 | 2.9 | 100.0 |
| 647 | 3.2 | 99.6 |
| 657 | 3.56 | 99.6 |
| 665 | 4.08 | 99.6 |
| 623 | 4.74 | 99.5 |
| 629 | 5.77 | 99.5 |
| 620 | 7.47 | 99.4 |
| 630 | 8.90 | 99.3 |
| 614 | 11.20 | 99.0 |
| 598 | 14.20 | 98.3 |
| 523 | 17.90 | 91.8 |
| 9 | 22.40 | 3.7 |
| 2 | 28.40 | 1.3 |
| 1 | 31.70 | 0.7 |

TABLE IIa
COKE FLOUR 3020

| | |
|---|---|
| Sample - Coke Flour 3020 Electrolyte - .9% NaCl-H₂O Wt of Sample - .001g \pm .0003 | Aperture - 200 μ Measuring Vol - 2000 μl Total Vol - 250ml |
|---|---|

| <u>n</u> | <u>d</u> | <u>% Above Stated Size</u> |
|----------|----------|--------------------------------|
| 15,197 | 5.2 | 100.0 |
| 10,748 | 5.6 | 97.8 |
| 7,866 | 6.2 | 95.9 |
| 5,965 | 7.1 | 94.1 |
| 4,177 | 8.1 | 91.5 |
| 2,639 | 9.6 | 88.1 |
| 1,593 | 11.8 | 83.8 |
| 915 | 14.6 | 78.8 |
| 543 | 18.0 | 74.4 |
| 317 | 23.0 | 67.0 |
| 176 | 29.0 | 59.0 |
| 107 | 36.0 | 51.3 |
| 57 | 46.0 | 31.2 |
| 23 | 57.0 | 24.9 |
| 14 | 64.0 | 18.5 |
| 9 | 69.0 | 13.8 |
| 6 | 74.0 | 10.3 |
| 3 | 78.0 | 6.2 |
| 1 | 87.5 | 2.5 |

TABLE IIb
SIEVE ANALYSIS OF 3020

Sample - Coke Flour 3020
Sieve Analysis

| <u>μ</u> (microns) | <u>Sieve Screen</u> | <u>% Sample</u> |
|-----------------------------------|---------------------|-----------------|
| | 35 | |
| 208 | 65 | 0.4 |
| 147 | 100 | 6.0 |
| 104 | 150 | 24.2 |
| 73 | 200 | 13.6 |
| 53 | 270 | 20.0 |
| | Pan | 35.8 |

TABLE IIIa
COKE FLOUR 3015

| | |
|---|---|
| Sample - Coke Flour 3015 | Aperture - 200 μ |
| Electrolyte - .9% NaCl-H₂O | Measuring Vol - 2000 μl |
| Wt of Sample - .001g \pm .0003g | Total Vol - 250ml |

| <u>n</u> | <u>d</u> | <u>% Above Stated Size</u> |
|----------|----------|----------------------------|
| 8905 | 5.2 | 100.0 |
| 6343 | 5.6 | 97.9 |
| 4500 | 6.2 | 95.7 |
| 3133 | 7.1 | 93.5 |
| 2223 | 8.1 | 91.3 |

TABLE IIIa (Cont'd)

COKE FLOUR 3015

| | |
|---|------------------------------|
| Sample - Coke Flour 3015 | Aperture - 200 μ |
| Electrolyte - .9% NaCl-H ₂ O | Measuring Vol - 2000 μ l |
| Wt of Sample - .001g \pm .0003g | Total Vol - 250ml |

| <u>n</u> | <u>d</u> | <u>% Above Stated Size</u> |
|----------|----------|--------------------------------|
| 1357 | 9.6 | 88.0 |
| 807 | 11.8 | 84.3 |
| 464 | 14.6 | 79.9 |
| 261 | 18.0 | 74.9 |
| 165 | 23.0 | 70.2 |
| 68 | 29.0 | 60.9 |
| 46 | 36.0 | 56.7 |
| 29 | 46.0 | 51.1 |
| 23 | 57.0 | 46.5 |
| 16 | 64.0 | 38.0 |
| 15 | 69.0 | 36.2 |
| 9 | 74.0 | 26.4 |
| 10 | 78.0 | 24.0 |
| 5 | 88.0 | 8.7 |
| 3 | 99.0 | - |

TABLE IIIb
SIEVE ANALYSIS OF 3015

Sample - Coke Flour 3015
Sieve Analysis

| <u>μ</u> (microns) | <u>Sieve Screen</u> | <u>% Sample</u> |
|-----------------------------------|---------------------|-----------------|
| | 35 | |
| 208 | 65 | 0.4 |
| 147 | 100 | 6.0 |
| 104 | 150 | 24.2 |
| 73 | 200 | 13.6 |
| 53 | 270 | 20.0 |
| | Pan | 35.8 |

TABLE IV
REYNOLDS 1-131 Al DUST

| | |
|--|--|
| Sample - Reynolds 1-131 Al Electrolyte - .9% NaCl-H ₂ O Wt of Sample - .001g \pm .0003g | Aperture - 140 μ Measuring Vol - 500 μ l Total Vol - 250ml |
|--|--|

| n* | n** | d | % Above Stated Size | |
|------|------|------|---------------------|-------|
| | | | a | b |
| 3829 | 2417 | 2.9 | 100.0 | 100.0 |
| 3581 | 2135 | 3.2 | 99.6 | 99.2 |
| 3143 | 1869 | 3.56 | 98.7 | 98.1 |
| 2604 | 1586 | 4.08 | 97.0 | 96.6 |
| 2272 | 1336 | 4.74 | 95.5 | 94.6 |
| 1647 | 985 | 5.77 | 90.8 | 89.9 |
| 1042 | 660 | 7.47 | 82.1 | 81.4 |
| 606 | 368 | 8.9 | 69.9 | 67.0 |
| 293 | 155 | 11.2 | 52.8 | 46.4 |
| 117 | 58 | 14.2 | 33.7 | 27.6 |
| 29 | 12 | 17.9 | 14.7 | 10.0 |
| 4 | 1 | 22.4 | 4.0 | 1.5 |
| 1 | 0 | 28.4 | 1.4 | 0.0 |
| 0 | 0 | 31.7 | - | - |

n* Run a
n** Run b

TABLE V
REYNOLDS 1-131 Al DUST

Sample - Reynolds 1-131 Al
Electrolyte - 4% NH₄CNS-
isopropanol
Wt of Sample - .001g \pm .0003g

Aperture - 140 μ
Measuring Vol - 500 μ l
Total Vol - 250ml

| n* | n** | d | % Above Stated Size | |
|------|------|------|---------------------|-------|
| | | | a | b |
| 2929 | 4339 | 2.04 | 100.0 | 100.0 |
| 2498 | 4001 | 2.3 | 99.6 | 99.8 |
| 2284 | 3649 | 2.5 | 99.3 | 99.5 |
| 2029 | 3270 | 2.9 | 98.9 | 99.0 |
| 1829 | 2911 | 3.4 | 98.3 | 98.4 |
| 1637 | 2468 | 4.2 | 97.5 | 97.1 |
| 1206 | 1893 | 5.1 | 93.9 | 93.9 |
| 911 | 1367 | 6.3 | 89.1 | 88.1 |
| 549 | 815 | 8.0 | 77.7 | 76.3 |
| 298 | 388 | 10.1 | 62.9 | 58.1 |
| 115 | 179 | 12.8 | 40.1 | 40.3 |
| 34 | 51 | 16.0 | 17.9 | 18.9 |
| 7 | 7 | 20.1 | 5.9 | 4.0 |
| 1 | 1 | 22.6 | 1.1 | 0.8 |
| 0 | 0 | 25.3 | - | - |

n* Run a
n** Run b

TABLE VI

MICROSCOPIC SCANNING

Sample - Reynolds 1-131 Al Dust
Objective - 43X
Filar Eye Piece

| n | d | Cum Wt % | n | d | Cum Wt % |
|----|------|----------|----|------|----------|
| 9 | > 30 | 4.6 | 2 | > 35 | .65 |
| 5 | 28 | 7.2 | 7 | 30.5 | 5.0 |
| 11 | 23 | 12.9 | 56 | 20.5 | 21.4 |
| 16 | 18.5 | 21.1 | 69 | 13 | 45.3 |
| 17 | 15.5 | 29.9 | 82 | 8 | 73.0 |
| 15 | 13.5 | 37.6 | 62 | 3.5 | 94.0 |
| 23 | 11.5 | 49.6 | 18 | < 2 | 100.0 |
| 28 | 9.5 | 64.0 | | | |
| 21 | 7.5 | 74.7 | | | |
| 22 | 5.5 | 86.1 | | | |
| 22 | 3 | 97.5 | | | |
| 5 | < 2 | 100.0 | | | |

TABLE VII
HYDRAULIC OIL MIXTURES

| <u>Electrolyte</u> | <u>%</u> | <u>% CH₂Cl₂</u> | | <u>% Trike</u> | <u>% Hydraulic Oil</u> |
|---------------------------|----------|---------------------------------------|----|----------------|------------------------|
| 20% LAI- | 25% | 25% | or | 25% | 50% |
| Isopropanol | 40% | 40% | or | 40% | 20% |
| 3-4% NH ₄ CNS- | 35% | | | 35% | 30% |
| Isopropanol | | | | | |
| .5% KCNS- | 30% | 30% | or | 30% | 40% |
| Acetone | 40% | 40% | | | 20% |
| | 20% | 50% | | | 30% |
| 4% ZnCl ₂ - | 25% | 25% | | | 50% |
| Acetone | | | | | |

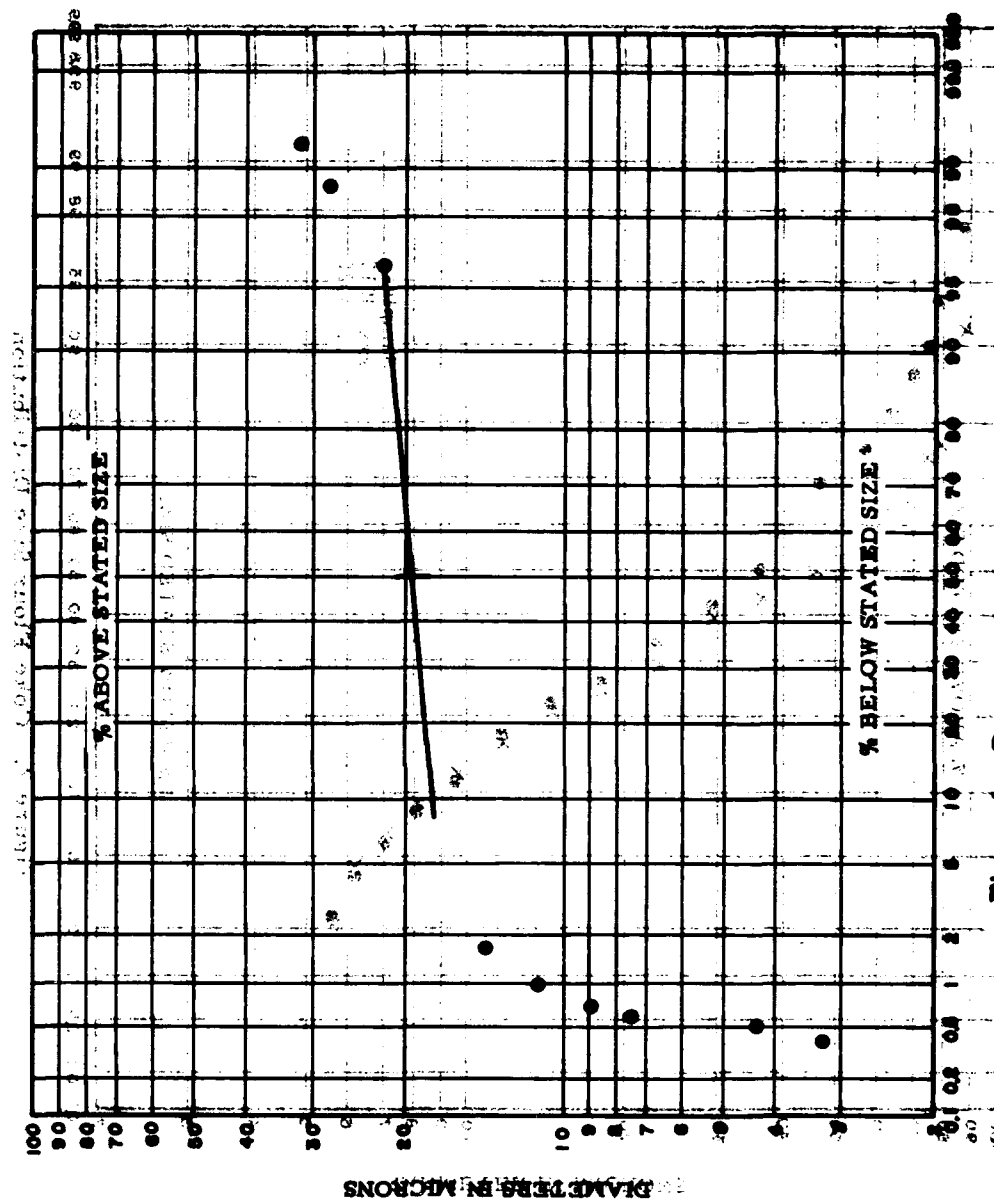


Figure 1. Ragweed Pollen Size Distribution

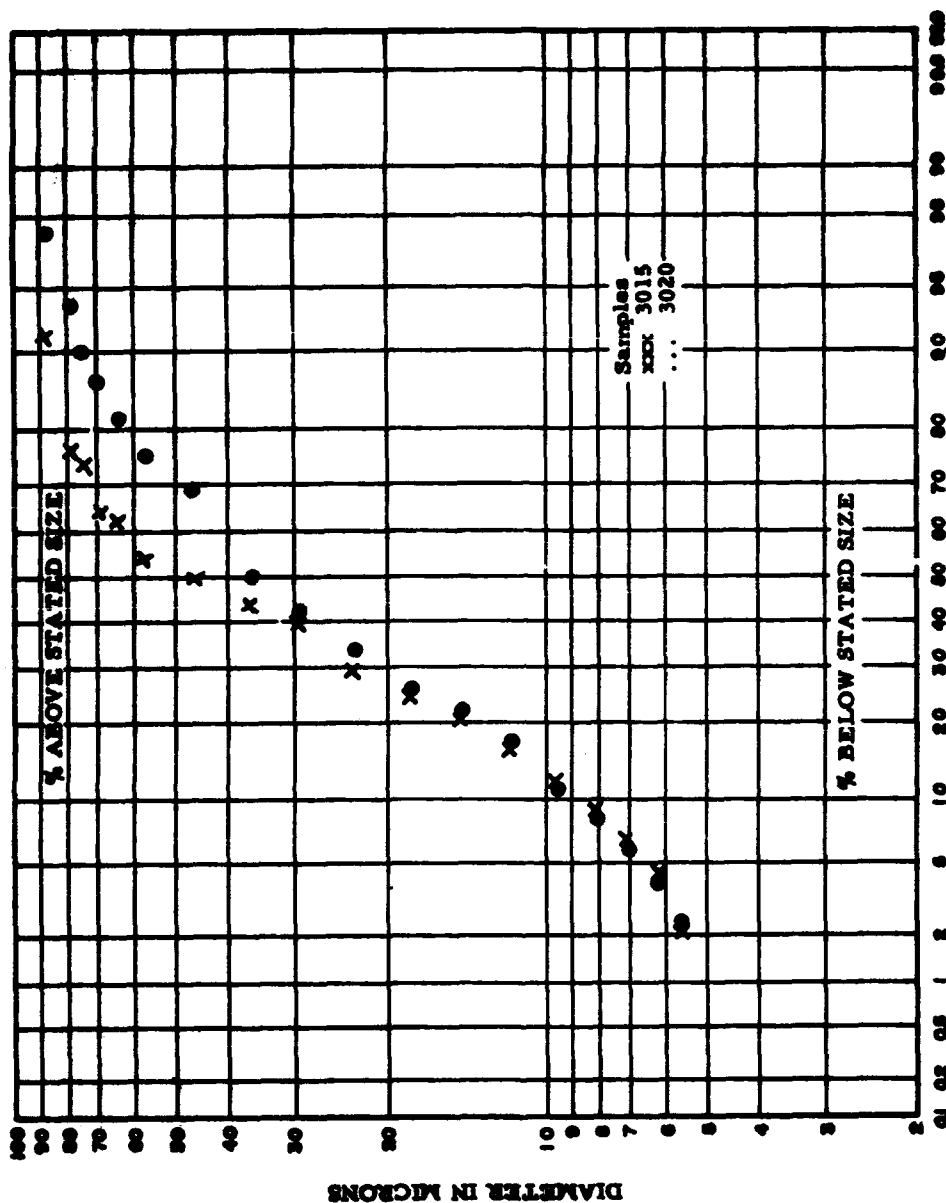


Figure 2. Coke Flour Size Distribution

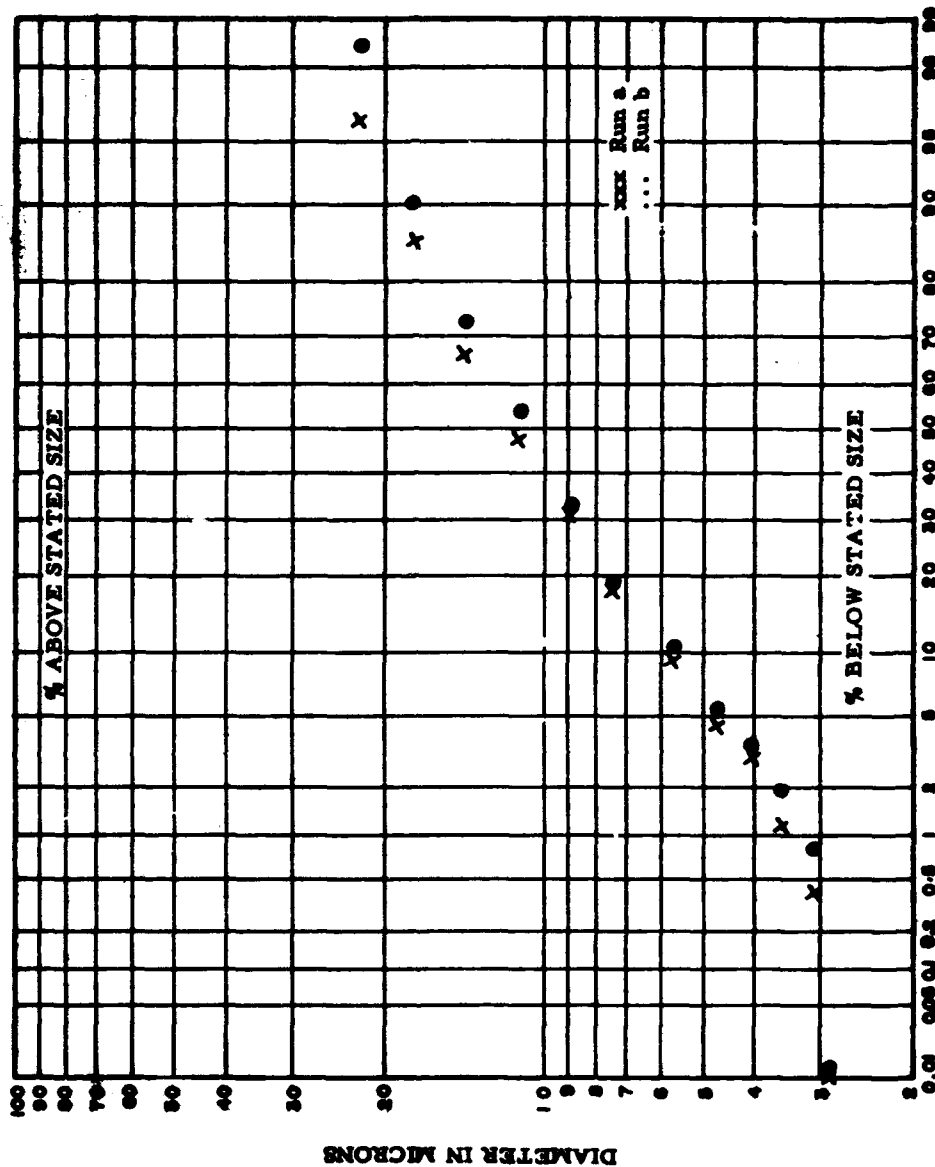


Figure 3. Size Distribution of Al 1-131 in 9% NaCl-H₂O

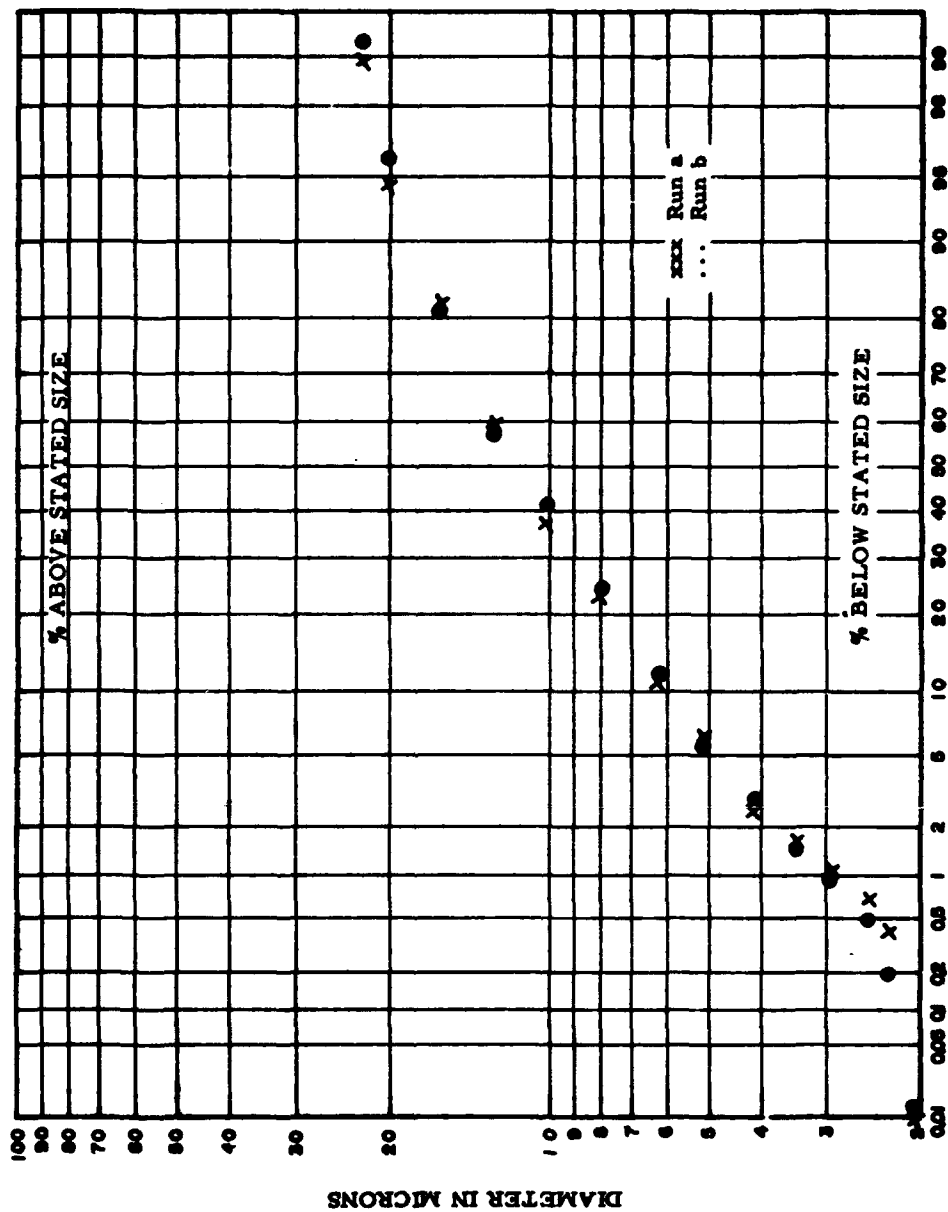


Figure 4. Size Distribution of Al 1-131 in 4% NH_4CNS -Isopropanol

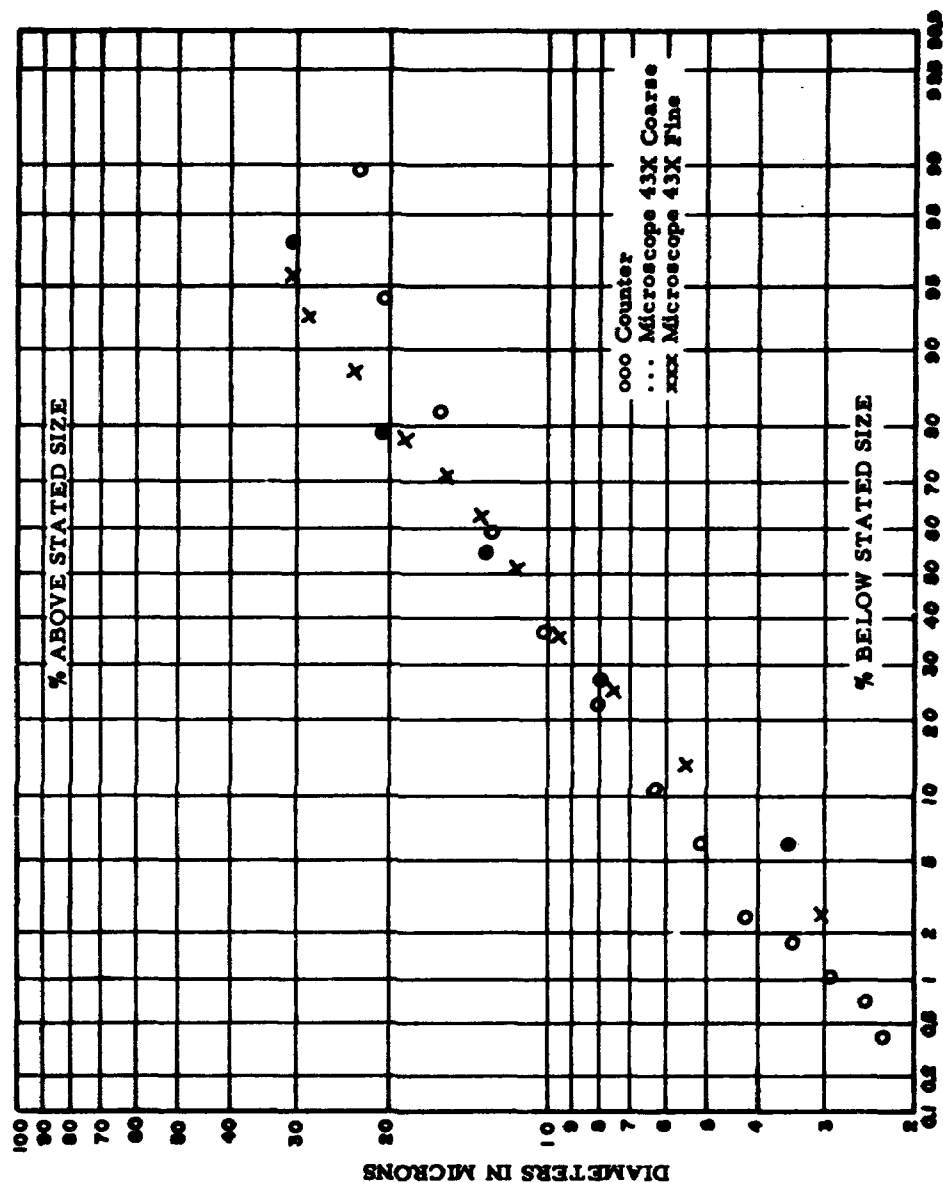


Figure 5. Counter VS. Microscopy Size Distribution of Al 1-131

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| 13. ABSTRACT <p>The objective of this study was to evaluate a method for the quality control of fine powders as an alternate method to that specified in MIL-P-27412 (USAF). The size distribution of various particles in the 3-100 micron range was studied using a counter which operates by the volumetric displacement of an electrolyte in an electrical sensing zone. The particle size distribution is affected by its compatibility with the electrolyte solution, and technique used to prepare the particles for sampling. Data obtained for size distribution was compared with data obtained by microscopic scanning, and from manufacturer's specifications.</p> <p>Analyses of aluminum powder, coke flour, and hydraulic oil, as well as the calibration sample ragweed pollen are given.</p> <p>Results show that the electrical sensing-zone counter can be used to give size distributions of a large number of 3-100 micron size particles with a good degree of accuracy, repeatability and speed.</p> | | |

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